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For the President of the European Patent Office

Le Président de l'Office européen des brevets
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Improvements relating to reactors

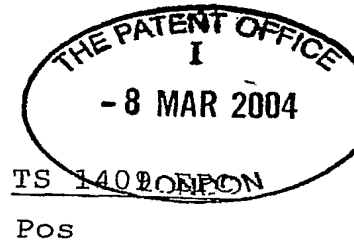
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IMPROVEMENTS RELATING TO REACTORS

The present invention relates to a filter system for use in a reactor. In particular, the invention relates to filter system suitable for a reactor to be used for exothermic reactions such as Fischer-Tropsch reactions.

5 The Fischer-Tropsch process is often used for the conversion of hydrocarbonaceous feed stocks into liquid and/or solid hydrocarbons. The feed stock (e.g. natural gas, associated gas, coal-bed methane and/or coal) is converted in a first step into a mixture of hydrogen and carbon monoxide (this mixture is often referred to as synthesis gas). The synthesis gas is then fed into a reactor where it is converted in a second step over a suitable catalyst at elevated temperature and pressure into paraffinic compounds ranging from methane to high molecular weight molecules comprising up to 200 carbon atoms, or, under particular circumstances, even more.

10 Numerous types of reactor systems have been developed for carrying out the Fischer-Tropsch reaction. For example, Fischer-Tropsch reactor systems include fixed bed reactors, especially multi tubular fixed bed reactors, fluidised bed reactors, such as entrained fluidised bed reactors and fixed fluidised bed reactors, and slurry bed reactors such as three-phase slurry bubble columns and ebulated bed reactors.

20 The Fischer-Tropsch reaction is very exothermic and temperature sensitive with the result that careful temperature control is required to maintain optimum

operation conditions and desired hydrocarbon product selectivity.

5 The heat transfer characteristics of fixed-bed reactors are generally poor because of the relatively low mass velocity, small particle size and low thermal capacity of fluids. If one attempts, however, to improve the heat transfer by increasing the gas velocity, a higher CO conversion can be obtained, but there is an excessive pressure drop across the reactor, which limits commercial viability. In order to obtain the CO conversions desired and gas through-puts of commercial interest, the conditions result in substantial radial temperature gradients. For that reason, the Fischer-Tropsch fixed-bed reactor tubes should have a diameter of less than 5 or 7 cm to avoid excessive radial temperature profiles. The desired use of high-activity catalysts in Fischer-Tropsch fixed-bed reactors makes the situation even worse. The poor heat transfer characteristics make local runaways (hotspots) possible, which may result in local deactivation of the catalyst. In order to avoid runaway reaction the maximum temperature within the reactor must be limited. However, the presence of temperature gradients within the reaction mixture means that much of the catalyst may be operating at sub-optimal levels.

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30 The use of liquid recycles as a means of improving the overall performance in a fixed-bed design has been described. Such a system is also called a "trickle bed" reactor (as part of a sub set of fixed-bed reactor systems) in which both reactant gas and liquid are introduced (preferably in an up flow or down flow orientation with respect to the catalyst) simultaneously. The presence of the flowing reactant gas and liquid

improves the reactor performance with respect to CO conversion and product selectivity. A limitation of the trickle bed system (as well as of any fixed-bed design) is the pressure drop associated with operating at high mass velocities. The gas-filled voidage in fixed-beds (typically less than 0.50) and size and shape of the catalyst particles does not permit high mass velocities without excessive pressure drops. Consequently, the mass through-put undergoing conversion per unit reactor volume is limited due to the head transfer rates. Increasing the individual catalyst particle size may slightly improve the heat transfer by allowing higher mass velocities (for a given pressure drop), but the loss in selectivity towards the high boiling point products and the increase in methane selectively combined with the increase in catalyst activity generally offset the commercial incentives of higher heat transfer.

Three-phase slurry bubble column reactors generally offer advantages over the fixed-bed design in terms of heat transfer characteristics. Such reactors typically incorporate small catalyst particles suspended by upward flowing gas in a liquid continuous matrix. In the case of multi-tubular reactors, the number of tubes incorporated is generally limited by mechanical parameters. The motion of the continuous liquid matrix allows sufficient heat transfer to achieve a high commercial productivity. The catalyst particles are moving within a liquid continuous phase, resulting in efficient transfer of heat generated from catalyst particles to the cooling surfaces, while the large liquid inventory in the reactor provides a high thermal inertia, which helps prevent rapid temperature increases that can lead to thermal runaway.

The present invention provides a filter system for use in a reactor, the filter system having filter means to separate fluid from a mixture of particulate matter and fluid, and having a filter housing adapted to connect to the reactor and adapted to receive the filter means, wherein the filter means is retractable into the housing.

The filter means is typically retractable through an aperture in the reactor, typically on an upper surface of the reactor, especially the top of a large tubular reactor as depicted in Figure 1. The housing is typically detachably connected to the aperture.

Typically the filter means has an export conduit leading from the filter means, to deliver filtrate from the filter means to processing equipment outside the reactor. The filter means may optionally have a connector for a hoist line, to facilitate retraction of the filter means into the housing, or simply may be retracted via the export conduit. The hoist line or the export conduit may be activated to pull the filter means into the housing from the reactor.

Typically the housing has an outlet for removing the filter means therefrom. In certain embodiment the housing can have an outlet port at the top or at a side through which the filter can be removed from the housing, or in more simple embodiments the housing is detachable from the reactor, so that the filter can be removed via the aperture used to retract the filter into the housing. A suitable housing is an elongated tube or pipe, preferably provided with detachable closing and/or sealing devices.

The housing preferably has at least one sealing device such as a valve for sealing the aperture between the reactor and the housing, so that when the filter means is retracted within the housing past the aperture,

the sealing device can be actuated to seal the aperture and isolate the housing from the reactor. This permits the subsequent removal of the filter means from the housing, and de-pressurisation of the housing, without de-pressurising the reactor.

Typically the filter means is lowered under gravity through an aperture in the upper surface of the reactor. When in use, the filters preferably are situated below the surface level of the expanded slurry bed.

In certain embodiments, the filter means can be provided with a cap adapted to prevent settling of fines on the filter means, and this is typically achieved by providing the cap with a sloped upper surface, so as to reduce the horizontal surface area presented by the filter means. In practice, conical or frusto-conical caps are preferred, with steep slopes so that fines of catalyst that settle on the top of the cap generally slide off and are dispersed into the slurry, and do not settle on the cap. This reduces the tendency of solid particles of catalyst to settle on the top of the filter means, which has the advantage that there is a reduced risk of uncontrolled exothermic reactions between the slurry and the localised build-up of catalyst on the top of the filter caps.

According to a further aspect, the invention provides a method of removing a filter from a reactor, the method comprising the steps of providing a filter housing communicating with the reactor through an aperture, retracting the filter from the reactor through the aperture and into the filter housing, and sealing the aperture between the reactor and the housing.

Typically the filter is removed from the housing after the aperture has been sealed, in order to maintain

the integrity of the pressure in the reactor. Optionally the housing can be pressurised to match the internal pressure of the reactor before the aperture to the reactor is opened, to replace the retracted filter with a new one.

~~Without wishing to be restricted to a particular~~
embodiment, the invention will now be described in further detail with reference to the drawings in which:

Figure 1 is a side view of a general arrangement of a reactor having a filter system;

Figure 2 is plan view of the reactor of Fig. 1, showing the arrangement of filters around the pipework of the Fig. 1 reactor;

Figure 3 is a side view of a filter system of the Fig. 1 reactor;

Figure 4 is a side view showing the detail of the filter module; and

Figure 5 is a cross-sectional view of a filter module.

Turning now to Figure 1 a reactor 20 has an outer shell 21 defining a chamber into which reactants are delivered. The reactor in this embodiment is typically used for carrying out three-phase slurry reactions, such as for example Fisher Tropsch type reactions.

Since the Fischer-Tropsch reaction is exothermic, the reactor 20 houses a number of cooling modules 1 for delivering and circulating coolant through the circulation system of coolant pipes within the reactor shell 21. Heat is transferred from the slurry surrounding the cooling modules 1 to the coolant as it passes through the circulation system of the module. Suitable coolants will be known to the person skilled in the art and include for example water/steam or oil based coolants.

Liquid phase reactants and solid particulate catalyst are fed into the reactor chamber from feed pipes (not shown), and gas phase reactants are delivered at the base of the reactor. The bubbles of gas rise in the liquid phase and interact with the solid particulate catalyst in the liquid phase to form reaction products, which are removed from the reactor. Gas phase products are typically removed by cyclones (not shown), and the light wax fractions that leave the reactor in gas phase can be recovered by cooling/condensation. The heavier fractions largely composed of liquid waxes comprising high-value long-chain hydrocarbons are separated from the slurry by filtration before being recovered for further processing.

The filter system comprises a number of filter modules 30, typically arranged around the densely packed cooling modules 1 (shown in outline in Fig. 2) preferably around the periphery of the array of cooling modules 1. 8 filter modules 30 are shown in the Fig. 1 embodiment, but the number of filter modules used can vary in accordance with the circumstances. Typically 2-20, preferably 4-10, filter modules 30 would be advantageous. In a preferred embodiment the filters are used as in pairs of filter arranged in a vertical assemblment, i.e. one filter immediately above another filter. In this situation a relative small housing can be used.

The type of filter selected is not crucial to the invention, but in this embodiment, each filter module 30 comprises a pair of filter bundles 31 formed from woven stainless steel gauze material. A useful configuration is a "sandwich" construction achieved by sintering the layers together, and the resultant material is then formed into filter tubes 32 of OD 40 mm and length 4 m, of which about 50-60 are arranged in each filter

bundle 31 around a central export conduit 33. Each filter module 30 can comprise one or more filter bundles 31 connected in series or in parallel to a common export conduit 33. One filter module 30 is shown in the drawings.

The export conduit 33 carries the filtered wax products through an outlet of the reactor for further processing, and optionally supports the weight of the filter module while it is suspended in the reactor.

The export conduit 33 passes intact through a flanged aperture 35 in the upper surface of the reactor 20 and through a filter housing 38 attached to the flanged aperture 35. The export conduit 33 passes intact through an iris gland at the top of the filter housing 38. The filter housing 38 is disposed directly above the flanged aperture and is connected to the flange via a valve 39, which can be closed to seal the aperture and thereby isolate the reactor 20 from the filter housing 38. Filtered products passing through the export conduit 33 are passed to further processing equipment P.

The filter housing 38 is in the form of a long cylinder slightly longer and wider than the filter modules 30, so that a single filter module 30 can be received within the housing 38 without obstructing the subsequent sealing of the valve 39.

In operation, the filter modules 30 hang below the apertures 35, and are suspended from the export lines 33 in the upper part of the reactor, but immersed in the slurry, as shown on the right hand side of Fig. 1. In normal operation, a layer of catalyst builds up on the surface of the filter modules, and this helps the filtration process as clean wax is separated through the pores of the catalyst layer. The flow rate of wax through

the export conduit 33 is controlled by creating and adjusting a pressure differential across the filter module 30. Continuous filtration can be achieved at various flow rates using appropriate pressure differentials, but those skilled in the art will

~~appreciate the rates and pressure differentials may be~~
varied in accordance with the filter type, surface area, viscosity of the fluids, and the various other characteristics of the system.

The location of the filter modules 30 at the top of the slurry zone in this embodiment gives various advantages. Notably, the local concentration of catalyst at the top of the slurry zone is less than at the bottom of the reactor, due to the tendency of the catalyst particles to settle under gravity in the slurry. This reduction of particulate matter in the area of the filter modules 30 reduces their tendency to block, and increases their useful life. In addition, filter modules 30 will displace cooling modules from the reactor space there is a risk of uncontrolled reactions occurring adjacent to the filter modules 30. Therefore, it is useful to site the filter modules 30 in an area of the slurry zone where the reactions can be limited by the naturally reduced concentrations of catalyst particles. Also, locating the filter modules 30 at the top of the slurry zone minimises the required length of the filter modules, and the export conduit 33 or hoist apparatus, thereby facilitating handling of the filter modules 30 during changeovers.

When a filter module 30 becomes blocked or damaged, or when the filtration rate falls, the pressure differential across the module 30 can be reversed to blow waxes or cleaning fluids back into the filter to clear the blockage. This can be conducted regularly as part of

the normal operating procedures, and may improve the filtration properties of the module in question, but at some point the filter module 30 will need to be removed from the reactor 20 for repair or replacement.

5 When a filter module is to be removed, the gas
~~injection into the reactor is optionally interrupted, the~~
pressure differential across the module 30 is removed or
reduced, and the export conduit 33 is withdrawn through
the iris gland at the top of the filter housing 38 to
10 retract the filter module 30 through the aperture at the
top of the reactor 20 and into the filter housing 38, as
shown on the left hand side of Fig. 1. At this point the
pressure in the filter housing 38 is equalised with the
pressure in the reactor. When the filter module 30 is
15 wholly within the housing 38, the valve 35 is closed to
seal off the aperture and isolate the filter housing 38
from the reactor 20. At that point the filter housing 38
can be removed from the flanged aperture at the top of
the reactor entirely, and the filter module 30 removed
20 therefrom. Alternatively, the housing 38 can remain
attached to the reactor, and the filter module can be
removed through a port (not shown) through the end or
side wall of the housing 38. The damaged filter module 30
can then be repaired and a replacement or repaired filter
25 module 30 can be positioned within the housing 38, still
with the valve 35 being kept closed to isolate the
reactor 20 from the housing 38. When the replacement
filter module 38 is in place in the housing 38, and the
housing is secured to the flange, housing 38 can be
30 pressurised up to the reactor pressure, the valve 38 can
be opened again, and the replacement filter module can be
lowered into the slurry zone as shown in the right hand
side of Fig. 1.

This permits changeover of filter modules to repair or replace damaged or blocked filters without depressurising or cooling the reactor 20. The temporary cessation of gas injection is preferred (although not essential), and recovery of waxes can continue in certain circumstances through the remaining filters. Thus a rolling programme of filter replacement can be implemented without disturbing the production regime.

In some embodiments a number of export conduits can pass through a single aperture 35, to service more than one filter module 38 per aperture.

In some circumstances, fixed filters can also be incorporated into the reactor, typically at positions in the slurry zone below the removable filter modules 30.

The filter bundle 31 in each module 30 typically has a conical cap 31c with a steep slope in order to reduce the tendency of catalyst particles to settle on top of the filter bundles 31.

In certain circumstances a back-up external filtration system can be used, by diverting products flowing through the export conduit 33 into an external filtration loop, and back into the processing equipment P.

Modifications and improvements can be incorporated without departing from the scope of the invention. For example, the average particle size of the catalyst particles may vary between wide limits, depending *inter alia* on the type of slurry zone regime. Typically, the average particle size may range from 1 μm to 2 mm, preferably from 1 μm to 1 mm.

If the average particle size is greater than 100 μm , and the particles are not kept in suspension by a mechanical device, the slurry zone regime is commonly

referred to as ebulating bed regime. Preferably, the average particle size in an ebulating bed regime is less than 600 μm , more preferably in the range from 100 to 400 μm . It will be appreciated that in general the larger the particle size of a particle, the smaller the chance
5 ~~that the particle escapes from the slurry zone into the~~
freeboard zone. Thus, if an ebulating bed regime is employed, primarily fines of catalyst particles will escape to the freeboard zone.

10 If the average particle size is at most 100 μm , and the particles are not kept in suspension by a mechanical device, the slurry zone regime is commonly referred to as a slurry phase regime. Preferably, the average particle size in a slurry phase regime is more than 5 μm , more
15 preferably in the range from 10 to 75 μm .

If the particles are kept in suspension by a mechanical device, the slurry zone regime is commonly referred to as stirred tank regime. It will be appreciated that in principle any average particle size
20 within the above ranges can be applied. Preferably, the average particle size is kept in the range from 1 to 200 μm .

The concentration of catalyst particles present in the slurry may range from 5 to 45% by volume, preferably,
25 from 10 to 35% by volume. It may be desired to add in addition other particles to the slurry, as set out in for example European Patent Application Publication No. 0 450 859. The total concentration of solid particles in the slurry is typically not more than 50% by volume,
30 preferably not more than 45% by volume. One or more draft tubes may be used to improve the catalyst dispersion.

Suitable slurry liquids are known to those skilled in the art. Typically, at least a part of the slurry liquid

is a reaction product of the exothermic reaction.
Preferably, the slurry liquid is substantially completely
a reaction product.

5 The exothermic reaction is a reaction which is
carried out in the presence of a solid catalyst, and
which is capable of being carried out in a three-phase
slurry reactor. Typically, at least one of the reactants
of the exothermic reaction is gaseous. Examples of
exothermic reactions include hydrogenation reactions,
10 hydroformylation, alkanol synthesis, the preparation of
aromatic urthanes using carbon monoxide, Kölbel-
Engelhardt synthesis, polyolefin synthesis, and Fischer-
Tropsch synthesis. According to a preferred embodiment of
the present invention, the exothermic reaction is a
15 Fischer-Tropsch synthesis reaction.

The Fischer-Tropsch synthesis is well known to those
skilled in the art and involves synthesis of hydrocarbons
from a gaseous mixture of hydrogen and carbon monoxide,
by contacting that mixture at reaction conditions with a
20 Fischer-Tropsch catalyst.

Products of the Fischer-Tropsch synthesis may range
from methane to heavy paraffinic waxes. Preferably, the
production of methane is minimised and a substantial
portion of the hydrocarbons produced have a carbon chain
25 length of a least 5 carbon atoms. Preferably, the amount
of C5+ hydrocarbons is at least 60% by weight of the
total product, more preferably, at least 70% by weight,
even more preferably, at least 80% by weight, most
preferably at least 85% by weight.

30 Fischer-Tropsch catalysts are known in the art, and
typically include a Group VIII metal component,
preferably cobalt, iron and/or ruthenium, more preferably
cobalt. Typically, the catalysts comprise a catalyst

carrier. The catalyst carrier is preferably porous, such as a porous inorganic refractory oxide, more preferably alumina, silica, titania, zirconia or mixtures thereof.

5 The optimum amount of catalytically active metal present on the carrier depends *inter alia* on the specific catalytically active metal. Typically, the amount of cobalt present in the catalyst may range from 1 to 100 parts by weight per 100 parts by weight of carrier material, preferably from 10 to 50 parts by weight per 100 parts by weight of carrier material.

10 The catalytically active metal may be present in the catalyst together with one or more metal promoters or co-catalysts. The promoters may be present as metals or as the metal oxide, depending upon the particular promoter concerned. Suitable promoters include oxides of metals from Groups IIA, IIIB, IVB, VB, VIB and/or VIIB of the Periodic Table, oxides of the lanthanides and/or the actinides. Preferably, the catalyst comprises at least one of an element in Group IVB, VB and/or VIIB of the Periodic Table, in particular titanium, zirconium, manganese and/or vanadium. As an alternative or in addition to the metal oxide promoter, the catalyst may comprise a metal promoter selected from Groups VIIB and/or VIII of the Periodic Table. Preferred metal promoters include rhenium, platinum and palladium.

25 A most suitable catalyst comprises cobalt as the catalytically active metal and zirconium as a promoter. Another most suitable catalyst comprises cobalt as the catalytically active metal and manganese and/or vanadium as a promoter.

30 The promoter, if present in the catalyst, is typically present in an amount of from 0.1 to 60 parts by weight per 100 parts by weight of carrier material. It

will, however, be appreciated that the optimum amount of promoter may vary for the respective elements which act as promoter. If the catalyst comprises cobalt as the catalytically active metal and manganese and/or vanadium as promoter, the cobalt : (manganese + vanadium) atomic ratio is advantageously at least 12:1.

The Fischer-Tropsch synthesis is preferably carried out at a temperature in the range from 125 to 350 °C, more preferably 175 to 275 °C, most preferably 200 to 260 °C. The pressure preferably ranges from 5 to 150 bar abs., more preferably from 5 to 80 bar abs.

Hydrogen and carbon monoxide (synthesis gas) is typically fed to the three-phase slurry reactor at a molar ratio in the range from 0.4 to 2.5. Preferably, the hydrogen to carbon monoxide molar ratio is in the range from 1.0 to 2.5.

The gaseous hourly space velocity may vary within wide ranges and is typically in the range from 1500 to 10000 Nl/l/h, preferably in the range from 2500 to 7500 Nl/l/h.

The Fischer-Tropsch synthesis is preferably carried out in a slurry phase regime or an ebulating bed regime, wherein the catalyst particles are kept in suspension by an upward superficial gas and/or liquid velocity.

It will be understood that the skilled person is capable to select the most appropriate conditions for a specific reactor configuration and reaction regime.

Preferably, the superficial gas velocity of the synthesis gas is in the range from 0.5 to 50 cm/sec, more preferably in the range from 5 to 35 cm/sec.

Typically, the superficial liquid velocity is kept in the range from 0.001 to 4.00 cm/sec, including liquid

production. It will be appreciated that the preferred range may depend on the preferred mode of operation.

According to one preferred embodiment, the superficial liquid velocity is kept in the range from
5 0.005 to 1.0 cm/sec.

C L A I M S

1. A filter system for use in a reactor, the
filter system having filter means to separate fluid from
a mixture of particulate matter and fluid, and having a
filter housing adapted to connect to the reactor and
5 adapted to receive the filter means, wherein the filter
means is retractable into the housing.
2. A filter system as claimed in claim 1, wherein the
filter means is retractable through an aperture in the
reactor.
- 10 3. A filter system as claimed in claim 1 or claim 2,
wherein the housing is typically detachably connected to
the aperture.
4. A filter system as claimed in any preceding claim,
wherein the filter means has an export conduit to deliver
15 filtrate from the filter means.
5. A filter system as claimed in any preceding claim,
wherein the filter means is retracted into the housing by
the export conduit.
6. A filter system as claimed in any preceding claim,
20 wherein the housing has an outlet for removing the filter
means therefrom.
7. A filter system as claimed in any preceding claim,
wherein the housing has at least one sealing device to
isolate the housing from the reactor.
- 25 8. A filter system as claimed in any preceding claim,
wherein the filter means has a cap adapted to prevent
settling of fines on the filter means.
9. A filter system as claimed in claim 8, wherein the
cap has a sloped upper surface.

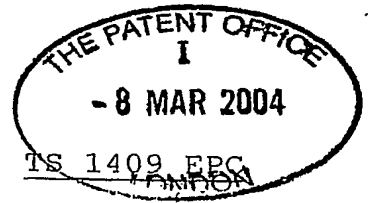
10. A method of removing a filter from a reactor, the method comprising the steps of providing a filter housing communicating with the reactor through an aperture, retracting the filter from the reactor through the aperture and into the filter housing, and sealing the aperture between the reactor and the housing.

11. A method according to claim 10, wherein the filter is removed from the housing after the aperture has been sealed.

12. A method according to claim 10 or claim 11, wherein the housing is pressurised to match the internal pressure of the reactor before the aperture to the reactor is opened.

13. A reactor provided with one or more filter system according to any of claims 1 to 8.

14. Process for the preparation of hydrocarbons in a reactor by reacting carbon monoxide and hydrogen in the presence of a catalyst, preferably a supported cobalt catalyst, and in the presence of liquid hydrocarbons, and removing formed liquid hydrocarbons from the reactor by a filter system according to any one or more of claims 1 to 9.



A B S T R A C T

IMPROVEMENTS RELATING TO REACTORS

A removable filter system for a reactor has a filter housing into which a filter module is retracted. The housing is sealed and the filter module removed without depressurising the reactor. This enables maintenance of filters without interrupting production from the reactor.

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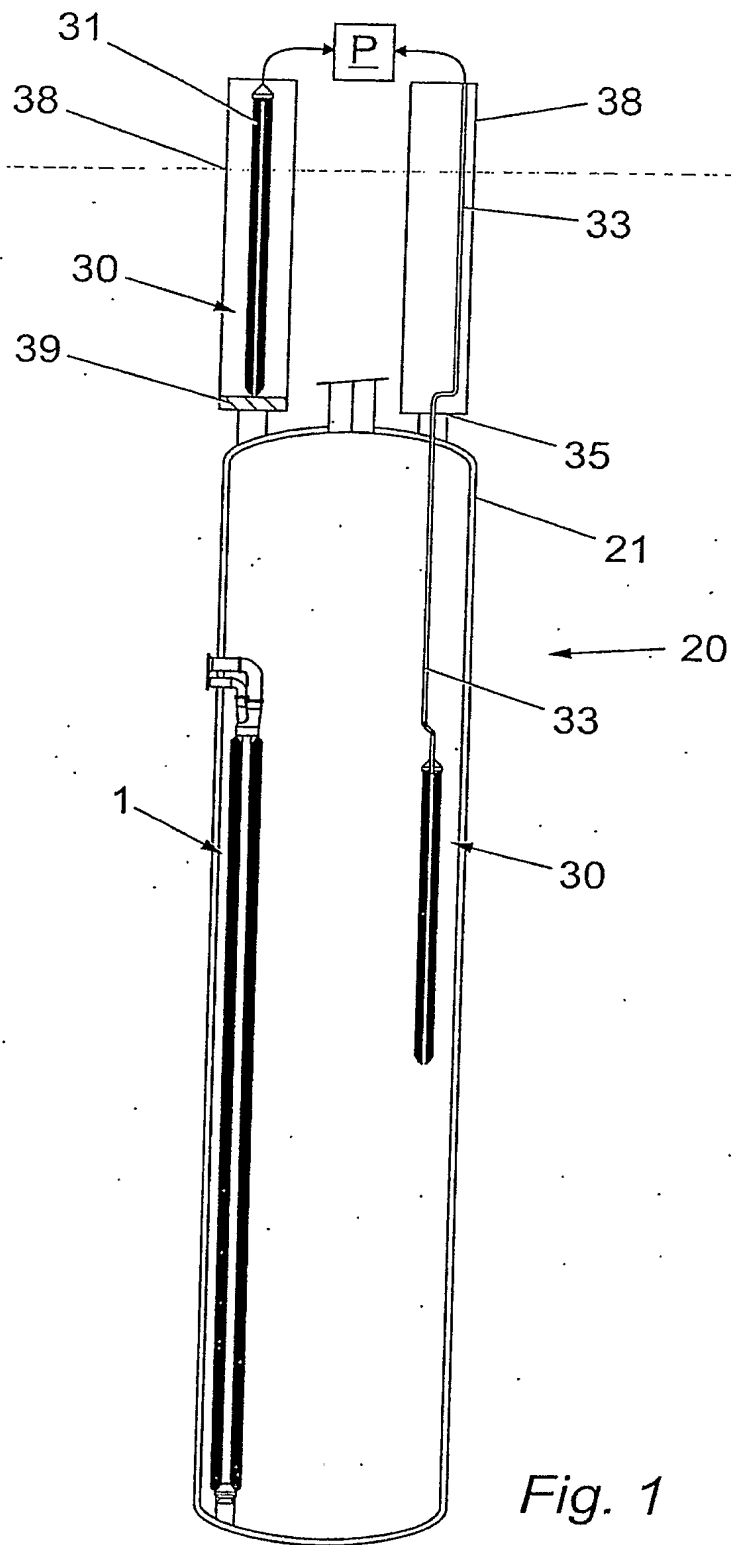


Fig. 1

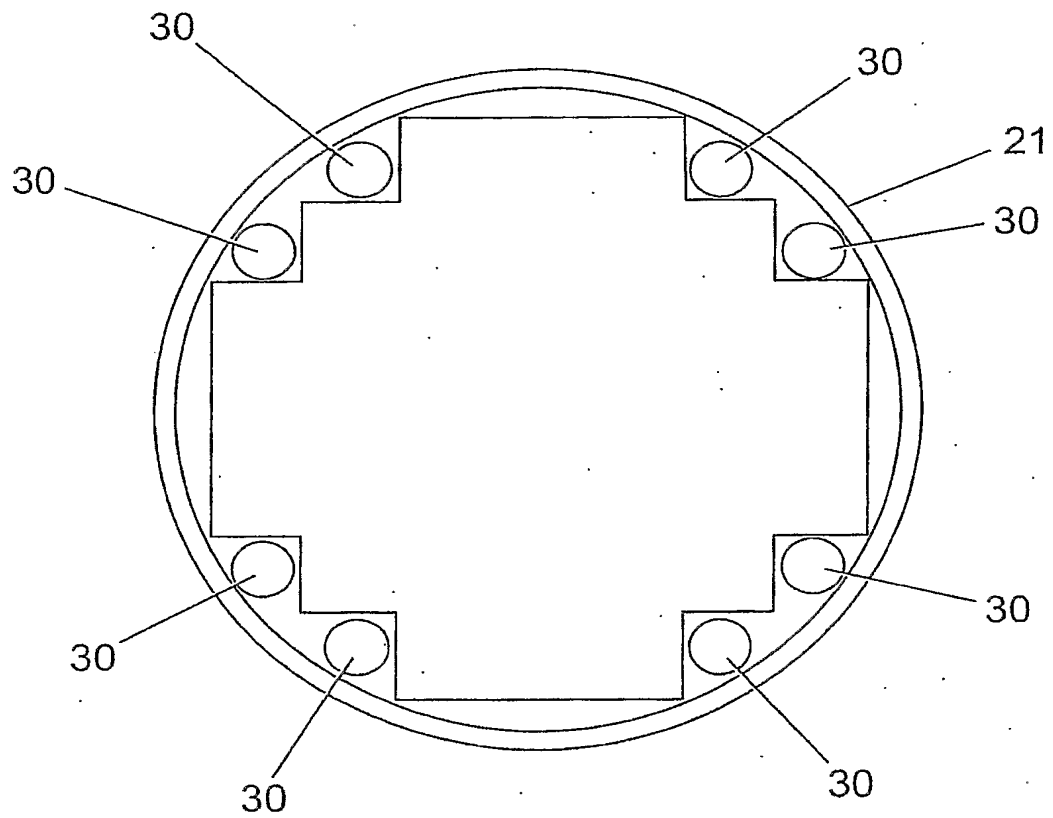


Fig. 2

